CLAIMS

- 1. A crystallization method of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine methyl ester crystals exhibiting the specific peaks of diffracted X-rays at angles of diffraction (20, CuK α rays) of at least 6.0°, 24.8°, 8.2° and 16.5°, which comprises using water alone or a mixture of water and a lower alcohol as the crystallization solvent and maintaining the nucleation temperature at 30°C or greater.
- 2. The crystallization method of Claim 1, wherein said lower alcohol is methanol, and the methanol content in the whole solution to be subjected to crystallization is 15 wt.% or less.
- 3. A crystallization method of $N-[N-(3,3-dimethylbutyl)-L-\alpha-aspartyl]-L-phenylalanine methyl ester crystals, which comprises using water alone or a mixture of water and a lower alcohol as the crystallization solvent and using, as the seed crystals <math>N-[N-(3,3-dimethylbutyl)-L-\alpha-aspartyl]-L-phenylalanine methyl ester crystals exhibiting the specific peaks of diffracted X-rays at angles of diffraction (20, CuK<math>\alpha$ rays) of at least 6.0°, 24.8°, 8.2° and 16.5°, whereby the same type of crystals as the seed crystals are allowed to be preferentially precipitated.
- 4. The crystallization method of Claim 3, wherein said lower alcohol is methanol.

